

## REPORT DOCUMENTATION PAGE

Form Approved  
OMB No. 0704-0188  
*(1)*

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED	
	June 15, 1994	Final Report 1 Sep 93-31 Aug 94	
4. TITLE AND SUBTITLE		5. FUNDING NUMBERS	
Scanning Tunneling Microscopy/Atomic Force Microscopy for Study of Nanoscale Metal Oxide Particles (Destructive Adsorbents)		DAAH04-93-G-0457	
6. AUTHOR(S)			
Kenneth J. Klabunde Dong Park			
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)		8. PERFORMING ORGANIZATION REPORT NUMBER	
Department of Chemistry Kansas State University Manhattan, Kansas 66506		<i>SP1100 SELECTED JUL 13 1994 SBD</i>	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)		10. SPONSORING / MONITORING AGENCY REPORT NUMBER	
U.S. Army Research Office P.O. Box 12211 Research Triangle Park, NC 27709-2211		ARO 32398.1-CH-DPS	
11. SUPPLEMENTARY NOTES The views, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.			
12a. DISTRIBUTION/AVAILABILITY STATEMENT		12b. DISTRIBUTION CODE	
Approved for public release; distribution unlimited.			
13. ABSTRACT (Maximum 200 words) A modern scanning tunneling microscopy/atomic force microscopy instrument was purchased. This instrument is allowing physical characterization of nanoscale particles of metal oxides. These particles are known to be effective reagents for the destruction of military toxins as well as chlorocarbons by a process we call destructive adsorption.			
DTIG QUALITY INSPECTED 6 <i>9P8</i> <b>94 7 12 060</b>			
14. SUBJECT TERMS Nanoscale particles, destructive adsorption, atomic force microscopy, scanning, tunneling, scanning probe		15. NUMBER OF PAGES 9	
		16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT UL

AD-A281 417

94-21306

Scanning Tunneling Microscopy/Atomic  
Force Microscopy for Study of Nanoscale  
Metal Oxide Particles (Destructive Adsorbents)

Final Report

June 14, 1994

U.S. Army Research Office

DAAHO4-93-0457

Kansas State University  
Department of Chemistry  
Manhattan, Kansas 66506

Approved for public release; distribution unlimited

Accession For	
NTIS GRA&I	<input checked="" type="checkbox"/>
DTIC TAB	<input type="checkbox"/>
Unannounced	<input type="checkbox"/>
Justification	
By _____	
Distribution _____	
Availability Codes	
Dist	Avail and/or Special
A-1	

## I. Introduction

The development of solid reagents that adsorb and simultaneously destroy toxic substances is important for air purification and as an alternative to incineration. Toxic chemicals such as organophosphorus, -halogen, -sulfur, and nitrogen compounds are not very amenable to treatment by high performance catalytic processes since these heteroatoms are notorious for catalyst poisoning.

Solid reagents that might serve as effective destructive adsorbents must have high capacity, and convert the toxic materials to non-toxic substances. Ideally, the heteroatoms (P, Cl, Br, N, S) would be extracted from the organic parts of the molecules and "mineralized" (converted to an innocuous metal salt).

In attempts to synthesize and develop such destructive adsorbents, we have concentrated our efforts on nanoscale metal oxides. The development of the most efficient solid reagents depends on our ability to prepare ultrafine powder with very high surface areas and intrinsic surface reactivities.

This research direction leads us into the exciting field of nanostructured materials, which possess novel and hybrid properties between molecular and bulk solid limits. Metal and semiconductor nanoscale materials have already been demonstrated to possess unique magnetic, optical, and physical properties. We are contributing to this field by studying the unique surface chemistry of nanoscale insulator particles.

From these points of view, we believe that continued work on the chemistry of such particles has importance both to basic and applied science. Further understanding of their synthesis, properties, and chemistry and their development into useful materials is the driving

force behind the prior and proposed work described herein.

As an important aspect of characterizing these nanoparticles, a scanning tunneling microscope/atomic force microscope. (STM/AFM) was purchased. This instrument allows observation of the particles before and after use as destructive adsorbents. Morphological properties such as surface defects are believed to be responsible for the chemical reactivity with toxic chemicals (chemical agents, chlorocarbons, etc.). In order to understand and correlate defect types and concentrations with reactivity, STM/AFM experiments are now in progress.

## **II. Instrument Purchased**

The STM/AFM instrument, model SPM 30, was purchased from WYCO corporation. The instrument consists of four major components: the microscope unit, the scanning probe interface, the stage/viewfinder controlled, and the computer system. To support the use of the microscope unit, a few necessary accessories were purchased, too. Those are two piezoelectric scanner modules of different scan-ranges, STM probe , AFM probe, probe holders, and cantilever carriers. An ink-jet printer and a color video copy processor were purchased from Hewlett Packard and Mitsubishi, respectively, to support the outcome of the resulting images.

## **III. Preliminary Results**

Three MgO powder samples with different surface area and surface morphology were studied by AFM. The surface area was 37, 241 and 325 m<sup>2</sup>/g, for samples A, B, and C, respectively. Those samples showed very different reactivity on the decomposition of chlorinated carbon components, which is speculated to be related to a different surface morphology. The fact that the samples were very fine powders gave a few technical difficulties in carrying out the AFM measurements, which were overcome by pressing the powder into a pellet at a pressure of 12000 psi. Figure 1 shows the surface morphology of the samples obtained by AFM. As apparent from the micrographs, samples have dramatically different surface morphology. Sample A consists of plates which are ~200 nm wide and ~50 nm thick stacked together along one direction. The alignment of the plates is speculated to be caused by pressure applied during the preparation of the pellet. The sample C consists of spheres of a diameter of ~100 nm. The surface of the sample C is rougher than sample A. Sample B is rather similar to A, though the sample also had a feature of sample C: in some section of the pellet, spheres were observed mixed with the plates. These AFM observations are consistent with TEM studies carried out previously on similar samples, which revealed two different kinds of particles: sphere and plates. The difference in the morphology suggests that the identity of the most exposed crystalline facet and possibly of defects are not identical for the MgO samples. An effort to try to identify the facets of the particles is underway by carrying out the AFM measurements at atomic resolution.

#### **IV. Students, Postdoctorals, and Research Associates Involved**

**Olga Koper, current Ph.D. student**

**Dong Park, postdoctoral research associate**

**Shawn Decker, current Ph.D. student**

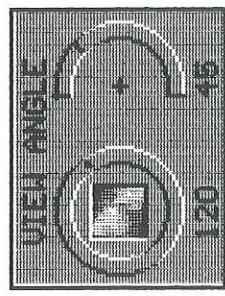
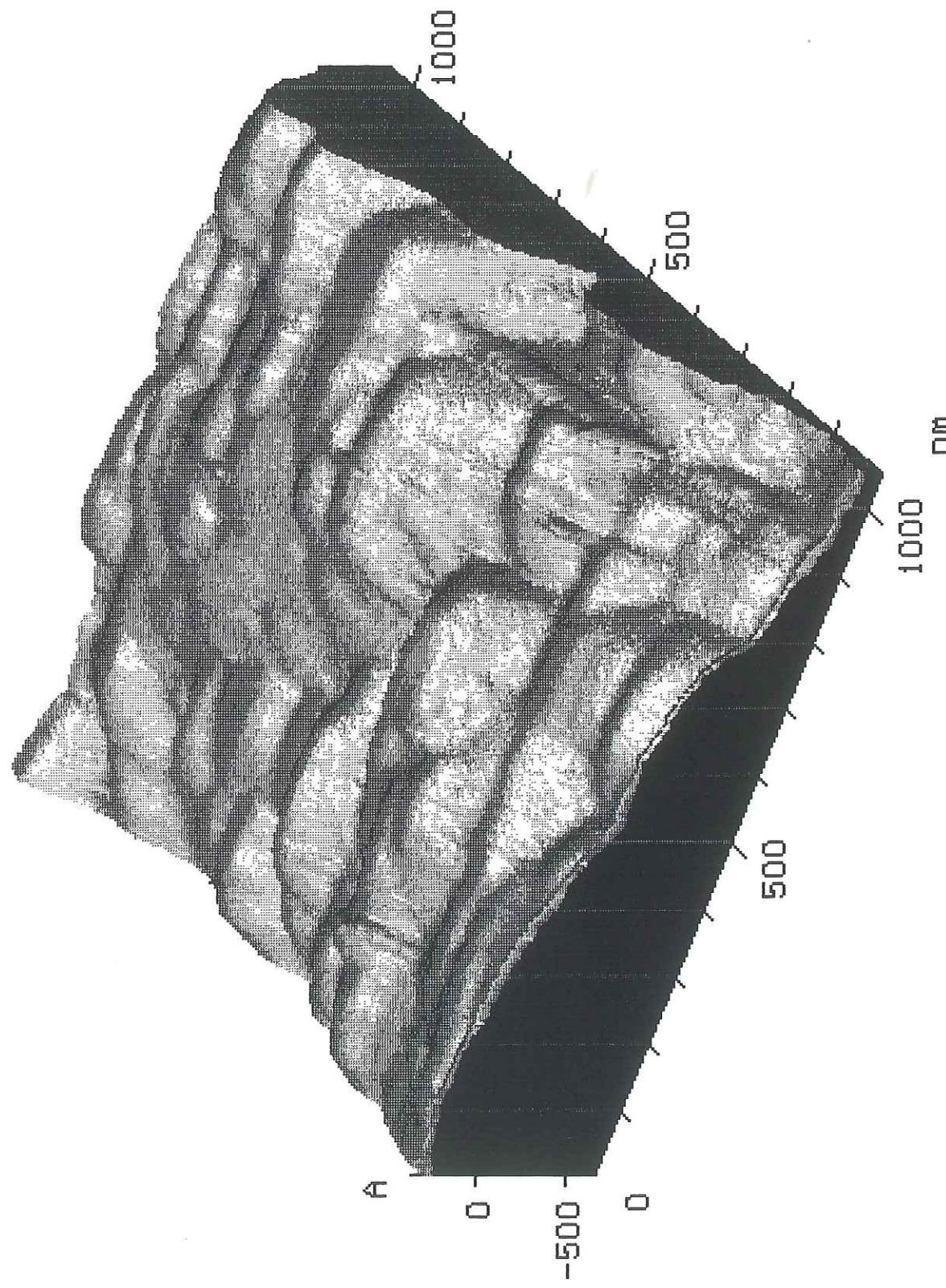
**Yan Jiang, current M.S. student**

New (17:31:54)

# Surface Plot

May 23, 1994

17:31:54



33 HK40.AFFM

Time: 13:04

Jun 06, 1994

Zoom: 1.00

WYKO SPM

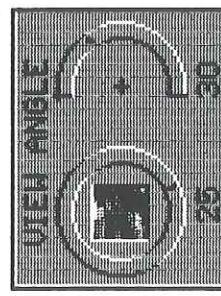
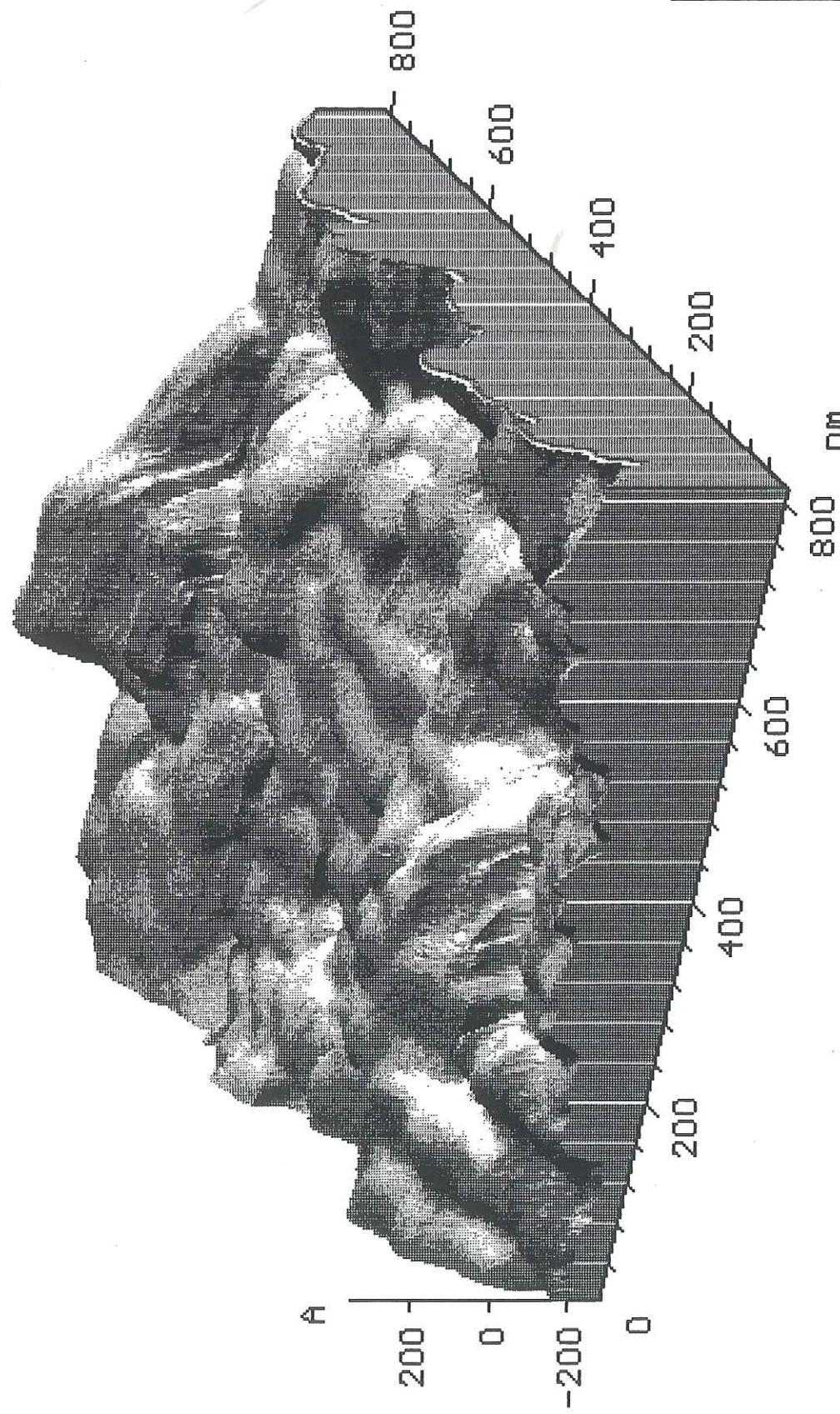
B

New (08:24:30)

# Surface Plot

08:24:30

May 06, 1994



>> Rk4D\_AFM.R

Time: 08:03

May 06, 1994

Zoom 1.00

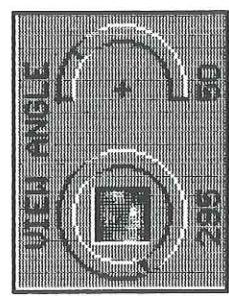
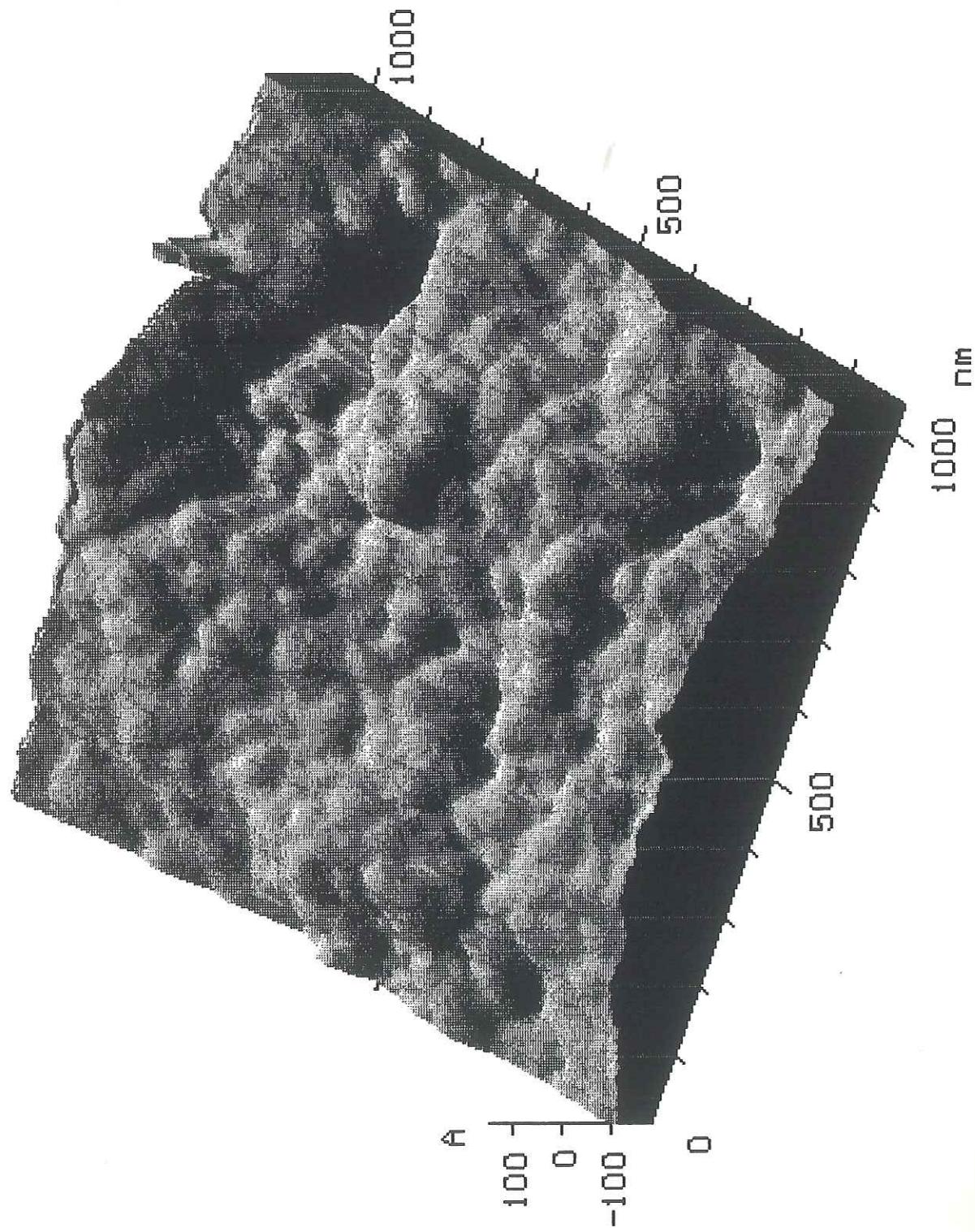
WYKO SPM

New (14:03:16)

# Surface Plot

14:03:16

File 09.1994



YY\_Rk40.affm& Zoom: 1.00 Timer: 13:27 300.06, 1920 14400 SPM